COMPOSITE MATERIALS COMPRISING A REINFORCING MATERIAL AND A THERMOPLASTIC MATRIX, PRECURSOR COMPOUND ARTICLE OF SAID MATERIALS AND PRODUCTS OBTAINED USING SAME

5 The field of the invention is that of composite materials and their manufacturing processes.

More precisely, the invention relates to the use of certain polycondensates for impregnating reinforcing 10 materials, especially in the form of yarns and/or fibers, which are intended to act as thermoplastic matrix in composite materials.

The term "yarn" is understood to mean a monofilament, a continuous multifilament yarn or a staple fiber yarn, obtained from a single type of fiber or from several types of fiber as an intimate blend. The continuous yarn may also be obtained by assembling several multifilament yarns.

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The term "fiber" is understood to mean a filament or combination of filaments that are chopped, cracked or converted.

In the field of high-performance materials, composites have taken pride of place owing to their performance characteristics and the weight savings that they allow. The most common high-performance composites known to date are obtained from thermosetting resins, the use of which is limited to low-volume applications, mainly in the aeronautical industry, motor sports and, in the best cases, applications having manufacturing times of around 15 minutes, such as for example for the manufacture of skis. The cost of these materials and/or the

manufacturing times make them incompatible with high-volume usage.

One solution, with regard to the manufacturing time, is provided by composites having a thermoplastic matrix. Thermoplastic resins are in general known for their high impedes their use for impregnating viscosity, which reinforcing materials generally composed of very dense bundles of filaments. The use of commercially available thermoplastic, especially polyamide, matrices results in 10 impregnation difficulties requiring either prolonged impregnation times or high processing pressures. In most composite materials obtained from the matrices may have microvoids and unimpregnated regions. These microvoids cause the mechanical properties to drop, 15 it is aging of the material and, when premature laminated, delamination problems.

To improve the impregnation of reinforcing yarns with the 20 matrix and the adhesion between the reinforcing yarns and the matrix, several approaches have been explored.

The first of these approaches has consisted in using linear polyamides of lower molecular weight as matrix.

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Thus, document FR-2 158 422 describes a composite sheet consisting of a polyamide matrix and reinforcing fibers of the glass fiber type. The polyamide is obtained by the polycondensation of ε -caprolactam, the molecular weight of which is between 3000 and 25 000 g/mol, having the capability, thanks to its low viscosity, of suitably impregnating the reinforcing fibers and thus of limiting the appearance of microvoids in the finished product. That document also describes a process for forming this composite sheet.

In general, the use of low-molecular-weight polyamides in the matrix has the major drawback of impairing the mechanical properties of the composite, especially as regards tensile strength, resistance to elongation and fatigue behavior, since, when high-performance composites reinforced by long fibers are employed, the mechanical properties of these composites are dependent on the plasticity of the matrix, which transmits the stresses to the reinforcement, and on the mechanical properties of said matrix.

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Another approach for improving the impregnation of reinforcing fibers with the matrix consists in employing a matrix in the form of an oligomer or a prepolymer of low molecular weight which can be polymerized *in situ* by polycondensation.

Thus, document FR-A-2 603 891 relates to a process for manufacturing a composite material consisting of a polyamide matrix reinforced by long reinforcing fibers. 20 These fibers are impregnated with a polyamide prepolymer or oligomer which has, at each end of the molecular chain, a reactive functional group capable of reacting with another oligomer or prepolymer molecule under the effect of heating, which results in the extension of the 25 polymer chain in order to obtain a polyamide of high molecular weight. The oligomer or prepolymer of molecular weight has the characteristic of being fluid in the molten state. The polyamides used are preferably polyamides nylon-6, nylon-6,6, nylon-6,10, nylon-6,12, 30 nylon-11 and nylon-12. The impregnated fibers are then pultruded through a shaping die, at high temperature, so as to form profiles.

35 This process remains close to conventional polymerization processes and therefore has cycle times incompatible with

a high production rate. If the cycle times are adapted so as to make them compatible with mass production, the molecular weight of the polyamide obtained, which constitutes the matrix, is too low to provide the latter with a good level of mechanical properties.

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Document EP-B-0 133 825 describes a flexible composite material mainly consisting of a reinforcing material in the form of a roving of parallel continuous fibers impregnated with a thermoplastic powder, preferably a 10 polyamide powder, and of a thermoplastic matrix in the form of a sheath around the roving of continuous fibers, it being possible for this sheath to also be made of a polyamide. This material is characterized in that the constituent polymer of the thermoplastic matrix possesses 15 a melting point not exceeding that of the constituent polymer of the thermoplastic powder, so that sheathing of the powder-coated fibers is carried out by melting the thermoplastic matrix without melting powder, in such a way that the latter isolates the fibers 20 from the sheath.

One drawback of using a thermoplastic polymer in powder form is the need to use complex equipment, which limits the quantity of composite obtained. It is therefore clearly apparent that this process is not very compatible with mass production.

Document US-B-5 464 684 describes a hybrid yarn comprising a core of intimately mingled reinforcing filaments and low-viscosity polyamide filaments, which form the matrix. This core is covered with a continuous polyamide yarn, preferably of the same type as that used for the core. The polyamide used is of the nylon-6 or nylon-6,6 type, but may also consist of nylon-6,6 T, nylon-6,10, nylon-10 or a polyamide obtained from adipic

acid and 1,3-xylylenediamine. The reinforcing fibers are carbon fibers or glass fibers.

The technique used to manufacture such a hybrid yarn is admittedly suitable for low-volume applications, such as the manufacture of tennis rackets. However, it is difficult to envisage the use of such a method for production on a larger scale.

Document WO 03/029350 describes the use of a star polyamide as matrix, such a polyamide having a good melt flow index, allowing the reinforcing materials to be properly impregnated.

One object of the present invention is therefore to remedy the drawbacks described above by proposing a precursor article of a composite material, comprising various types of yarns and/or fibers, and especially at least one reinforcing yarn and/or fibers and at least one yarn and/or fibers generating a thermoplastic matrix having a high melt flow index, allowing very good impregnation of the reinforcing yarns and/or fibers during formation of the composite material. Such an article makes it possible to obtain a composite material using a simple rapid compression molding technique.

Another object of the invention is to propose a composite material obtained from this article and exhibiting good mechanical properties.

Lastly, a final object of the invention is to provide a composite material having the advantage of manufacturing cost reduction by the use of tooling operating at low pressures and with shorter cycle times.

For this purpose, the invention relates to a precursor article of a composite material comprising a polymeric matrix and at least one reinforcing yarn and/or fibers, said article comprising at least one reinforcing yarn and/or fibers and at least one polymeric-matrix yarn and/or fibers, characterized in that:

- said reinforcing yarn and/or fibers are made of reinforcing material and optionally include a part made of a thermoplastic polymer;
- said polymeric-matrix yarn and/or fibers are made of a thermoplastic polymer, and in that:
 - said thermoplastic polymer of said reinforcing yarn and/or fibers and/or of said polymeric-matrix yarn and/or fibers comprises at least one polycondensate consisting of:
 - 30 to 100 mol% (limits inclusive) of macromolecular chains satisfying the following formula (I):

20 $R_3 - (X - R_2 - Y)_n - X - A - R_1 - A - X - (Y - R_2 - X)_m - R_3$ (I)

• 0 to 70 mol% (limits inclusive) of macromolecular chains satisfying the following formula (II):

$$R_4 - [Y - R_2 - X]_p - R_3$$
 (II)

25 in which chains:

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- \bullet -X-, -Y- is a radical obtained from the condensation of two reactive functional groups F_1 and F_2 such that:
- F_1 is the precursor of the -X- radical and F_2 is 30 the precursor of the -Y- radical, or vice versa,
 - the functional groups $\ensuremath{F_{1}}$ cannot react together by condensation and
 - the functional groups F_2 cannot react together by condensation;

- A is a covalent bond or an aliphatic hydrocarbon radical that may comprise heteroatoms and contains 1 to 20 carbon atoms;
- ullet R₂ is a branched or unbranched, aliphatic or aromatic hydrocarbon radical containing 2 to 20 carbon atoms;
 - \bullet R₃, R₄ represents hydrogen, a hydroxyl radical or a hydrocarbon radical;
- ullet R₁ is a linear or cyclic, aromatic or aliphatic, 10 hydrocarbon radical containing at least 2 carbon atoms and possibly including heteroatoms; and
 - \bullet n, m and p each represent a number between 30 and 200.
- 15 All the known polycondensation functional groups may be used within the context of the invention for F_1 and F_2 .

In the polycondensate, the radicals R_2 may be of the same type or may differ from one another.

According to one particular embodiment of the invention, the polymeric matrix is a polyamide Al consisting of:

• 30 to 100 mol% (limits inclusive) of macromolecular chains satisfying the following formula (I):

$$R_3 - (X - R_2 - Y)_n - X - A - R_1 - A - X - (Y - R_2 - X)_m - R_3$$
 (I)

 \bullet 0 to 70 mol% (limits inclusive) of macromolecular chains satisfying the following formula (II):

$$R_4 - [Y - R_2 - X]_p - R_3$$
 (II)

30 in which:

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- Y is the R_s radical when X represents the -c- radical;

- -ç— ° radical when X represents the radical;
- A is a covalent bond or an aliphatic hydrocarbon radical possibly including heteroatoms and containing 1 to 20 carbon atoms;
 - R_2 is a branched or unbranched, aliphatic or aromatic, hydrocarbon radical containing 2 to 20 carbon atoms;
 - R_3 , R_4 represents hydrogen, a hydroxyl radical or a hydrocarbon radical comprising a $\stackrel{-c-}{\circ}$ or $\stackrel{N-}{R_s}$ group;
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 - R_5 represents hydrogen or a hydrocarbon radical containing 1 to 6 carbon atoms;
 - R_1 is a linear or cyclic, aromatic or aliphatic, hydrocarbon radical containing at least 2 carbon atoms and possibly including heteroatoms; and
 - n, m and p each represent a number between 30 and 200.
- According to another particular embodiment of invention, the polymeric matrix of the invention consists 20 of a polyester A2 consisting of:
 - 100 mol% (limits inclusive) 30 macromolecular chains satisfying the following formula (I):

 $R_3 - (X - R_2 - Y)_n - X - A - R_1 - A - X - (Y - R_2 - X)_m - R_3$ (I)

 0 to 70 mol% (limits inclusive) of macromolecular chains satisfying the following formula (II):

$$R_4 - [Y - R_2 - X]_p - R_3$$
 (II)

in which chains:

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- Y is the -O- radical when X represents the 30 radical;
 - Y is the oradical when X represents the -Oradical;

- A is a covalent bond or an aliphatic hydrocarbon radical possibly including heteroatoms and containing 1 to 20 carbon atoms;
- R_2 is a branched or unbranched, aliphatic or aromatic, hydrocarbon radical containing 2 to 20 carbon atoms;
 - R_3 , R_4 represents hydrogen, a hydroxyl radical or -c- a hydrocarbon radical comprising a or -O- group;
- R_1 is a linear or cyclic, aromatic or aliphatic, 10 hydrocarbon radical containing at least 2 carbon atoms and possibly including heteroatoms; and
 - n, m and p each represent a number between 30 and 200.
- 15 The polymeric matrix of the invention may also be a copolyesteramide.

Advantageously, m, n and p are between 30 and 250.

20 Advantageously, R_2 is a pentamethylene radical.

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The polyamide Al or the polyester A2 of the invention advantageously comprises at least 45 mol%, preferably at least 60 mol% and even more preferably at least 80 mol% of macromolecular chains satisfying formula (I).

The polyamide Al or the polyester A2 of the invention advantageously has a number-average molecular weight of not less than 5000 g/mol but not exceeding 25 000 g/mol.

The thermoplastic polymer yarns and/or fibers intended to act as matrix will be called hereafter "matrix yarns and/or fibers".

The term "number-average molecular weight" of the polyamide Al or of the polyester A2 is understood to mean the number-average molecular weight weighted by the molar fractions of the two types of macromolecular chain of formulae (I) and (II).

According to one particular embodiment of the invention, the polyamide Al or the polyester A2 is obtained by copolymerization from a monomer mixture comprising:

- a) a difunctional compound, the reactive functional groups of which are chosen from amines, carboxylic acids, alcohols and derivatives thereof, the reactive functional groups being identical;
- b) monomers of the following general formulae 15 (III $_a$) and (III $_b$) in the case of the polyamide A1:

$$X'-R'_2-Y'$$
 (III_a) or H (III_b)

b') monomers of the following general formulae (III $_a$ ') and (III $_b$ ') in the case of the polyester A2:

$$X'-R'_{2}-Y'$$
 (IIIa') or R'_{2} (IIIb')

20 in which formulae:

- R'₂ represents a substituted or unsubstituted, aliphatic, cycloaliphatic or aromatic hydrocarbon radical containing 2 to 20 carbon atoms and possibly including heteroatoms;
- Y' is an amine radical when X' represents a carboxylic radical, or Y' is a carboxylic radical when X'

represents an amine radical, in the case of the polyamide A1; and

• Y' is a hydroxyl radical when X' represents a carboxylic radical, or Y' is a carboxylic radical when X' represents a hydroxyl radical, in the case of the polyester A2.

The terms "carboxylic acid" and "carboxylic radical" are understood to mean in the present invention carboxylic acids and their derivatives, such as acid anhydrides, acid chlorides, esters, nitriles, etc. The term "amine" is understood to mean amines and their derivatives.

The monomers of formula (III_a) or (III_b) are preferably the monomers for polyamides of the type comprising nylon-6, nylon-11, nylon-12, etc. Examples of monomers of formula (III_a) or (III_b) that may be suitable in the context of the invention include caprolactam, 6-amino-caproic acid, lauryllactam, etc. A mixture of different monomers may also be used.

As examples of monomers of formula (III $_a$ ') or (III $_b$ ') that may be suitable within the context of the invention, mention may be made of caprolactone, δ -valerolactone, 4-hydroxybenzoic acid, etc.

The monomer mixture may also include a monofunctional monomer conventionally used in the production of polymers as a chain stopper.

The monomer mixture may also include catalysts.

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Advantageously, compound a) represents between 0.1 and 2 mol% relative to the number of moles of monomers of type b) or b').

In the case of the polyamide Al, the copolymerization of the monomers is carried out under conventional conditions for polymerizing polyamides obtained from lactams or amino acids.

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In the case of the polyester A2, the copolymerization of the monomers is carried out under conventional conditions for polymerizing polyesters obtained from lactones or hydroxyacids.

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The polymerization may include a finishing step so as to obtain the desired degree of polymerization.

embodiment particular another According to or the polyester A2 invention, the polyamide Al 15 obtained by melt blending, for example using an extruder, obtained those the type of polyamide of acids or and/or amino lactams polymerization of polyester of the type of those obtained by polymerization and a difunctional and/or hydroxyacids lactones 20 compound, the reactive functional groups of which are alcohols, carboxylic acids chosen from amines, derivatives thereof, the reactive functional groups being identical. The polyamide is for example nylon-6, nylonfor example polyester is The etc. 11, nylon-12, 25 polycaprolactone, polypivalolactone, etc.

The difunctional compound is added directly into the polyamide or the polyester in the melt state.

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Advantageously, the difunctional compound represents between 0.05 and 2% by weight relative to the weight of polyamide or polyester.

35 The difunctional compound of the invention is preferably represented by formula (IV):

$X''-A-R_1-A-X''$ (IV)

in which X" represents an amine radical, a hydroxyl radical, a carboxylic group or derivatives thereof, R_1 and A being as described above.

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Examples of the radical $X^{\prime\prime}$ that may be mentioned include a primary amine radical, a secondary amine radical, etc.

The difunctional compound may be a dicarboxylic acid.

Examples of diacids that may be mentioned include adipic acid, which is the preferred acid, decanoic or sebacic acid, dodecanoic acid and phthalic acids, such as terephthalic acid and isophthalic acid. It may be a mixture comprising by-products resulting from the manufacture of adipic acid, for example a mixture of adipic acid, glutaric acid and succinic acid.

The difunctional compound may be a diamine. Examples of diamines that may be mentioned include hexamethylenediamine, methylpentamethylenediamine, 4,4'-diamino-dicyclohexylmethane, butanediamine and meta-xylylenediamine.

The functional compound may be a dialcohol. Examples of dialcohols that may be mentioned include 1,3-propanediol, 1,2-ethanediol, 1,4-butanediol, 1,5-pentanediol, 1,6-hexanediol and polytetrahydrofuran.

The functional compound may be a mixture of a diamine and 30 a dialcohol.

In the case of the polyamide A1, the reactive functional groups of the difunctional compound are generally amines or carboxylic acids or derivatives thereof.

In the case of the polyester A2, the reactive functional groups of the difunctional compound are generally alcohols or carboxylic acids or derivatives thereof.

Preferably, the difunctional compound is chosen from adipic acid, decanoic or sebacic acid, dodecanoic acid, terephthalic acid, isophthalic acid, hexamethylenediamine, methylpentamethylenediamine, 4,4'-diamino-dicyclohexylmethane, butanediamine, metaxylylenediamine, 1,3-propanediol, 1,2-ethanediol, 1,4-butanediol, 1,5-pentanediol, 1,6-hexanediol and polytetrahydrofuran.

of embodiment another particular to According invention, the polyamide A1 or the polyester A2 obtained by melt blending, for example using an extruder, obtained those of the type polyamide of amino acids lactams and/or polymerization of polyester of the type of those obtained by polymerization lactones and/or hydroxyacids, with a compound of formula (V):

G-R-G (V)

in which:

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- R is a substituted or unsubstituted, linear or cyclic, aromatic or aliphatic hydrocarbon radical possibly including heteroatoms; and
- G is a functional group or a radical that may reactive with the amine either selectively reactive the alcohol with or functional groups, functional groups, or with the carboxylic acid reactive functional groups of the polyamide or of the polyester, in order to form covalent bonds. The polyamide is for example nylon-6, nylon-11 or nylon-12. The polyester is for example polycaprolactone or polypivalolactone.
- 35 The compound of formula (V) is added directly to the polyamide or to the polyester in the melt state.

Advantageously, the compound of formula (V) represents between 0.05 and 2% by weight relative to the weight of polyamide or polyester.

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All polymer chain couplers or polymer chain extenders known to those skilled in the art, generally comprising two identical functional groups or two identical radicals, and selectively reacting either with the amine reactive functional groups or with the alcohol reactive functional groups, or with the carboxylic acid reactive functional groups of the polyamide or of the polyester, in order to form covalent bonds, may be used as compound of formula (V).

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In the case of obtaining polyamide A1, compound (V) may for example react selectively with the amine functional groups of the polyamide into which it is introduced. This compound will not react with the acid functional groups of the polyamide in this case.

Advantageously, when the reinforcing yarns and/or fibers comprise a thermoplastic polymer, this is preferably in the form of a sheath of polymer that covers the reinforcing yarns and/or fibers.

According to a variant of the invention, the precursor article of the composite also includes at least one matrix yarn and/or fibers made of a linear thermoplastic polymer.

According to a preferred feature, this linear polymer is an aliphatic and/or semicrystalline polyamide or copolyamide chosen from the groups comprising nylon-4,6, nylon-6, nylon-6,6, nylon-6,9, nylon-6,10, nylon-6,12, nylon-6,36, nylon-11, nylon-12 or a semicrystalline

semiaromatic polyamide or copolyamide chosen from the group comprising polyphthalamides, and blends of these polymers and of their copolymers.

The matrix yarns and/or fibers may also include all the useful additives such as flame retardants, plasticizers, heat and light stabilizers, waxes, pigments, nucleating agents, antioxidants, impact modifiers or the like known to those skilled in the art.

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Advantageously, the reinforcing yarns and/or fibers are chosen from carbon, glass, aramid and polyimide yarns and/or fibers.

- 15 According to a variant of this feature, the reinforcing yarns and/or fibers are natural yarns and/or fibers chosen from sisal, hemp and flax yarns and/or fibers.
- Advantageously, the article according to the invention 20 also includes a matrix precursor powder material, which may for example be a polyamide.

Preferably, a powder having a particle size of between 1 and 100 microns will be used.

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Preferably, the article according to the invention is formed from continuous or chopped yarns, tapes, mats, braids, wovens, knits, webs, multiaxial fabrics, nonwovens and/or complex forms comprising several of the aforementioned forms. As examples, a complex form may be a web combined with a nonwoven or with continuous yarns.

Another subject of the invention is a composite obtained from an article as defined above by at least partial melting of the matrix yarns and/or fibers. This composite

comprises a polymeric matrix and reinforcing yarns and/or fibers.

The term "partial melting" is understood to mean the melting of at least part of at least one matrix yarn and/or fiber.

This melting may be carried out by compression molding by applying pressure at a temperature above the melting point of the polymeric matrix. This melting allows the reinforcing yarns and/or fibers to be uniformly impregnated with the matrix.

According to a preferred feature, the composite thus obtained has a reinforcement content of between 25 and 80% by weight.

Yet another subject of the invention is a semifinished product obtained by a process of compression molding or calendering the aforementioned article, during which the matrix yarns and/or fibers are at least partially melted so as to impregnate the reinforcing yarns and/or fibers.

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Also advantageously, this semifinished product is in 25 sheet or tape form.

The semifinished product consists of an intermediate product in which the reinforcing yarns and/or fibers have been impregnated with the polymeric matrix, which is in the form of a continuous phase. This product is not yet in its final form.

The semifinished product must undergo a subsequent forming step, by a forming or compression molding process known to those skilled in the art, at temperatures above

their glass transition temperature but below its melting point, allowing a finished product to be obtained.

Yet another subject of the invention is a finished product obtained by a process of compression molding the aforementioned article to the final shape, during which the matrix yarns and/or fibers are at least partially melted so as to impregnate the reinforcing yarns and/or fibers.

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In general, the compression molding processes used involve low pressures (below 20 bar), temperatures below 290°C and short times (less than 5 minutes).

- Other details and advantages of the invention will become more clearly apparent in the light of the examples given below, solely by way of indication and for the purpose of illustration.
- 20 Matrix used: polyamide A1 according to the invention, obtained by the polycondensation of caprolactam in the presence of 0.6 mol% adipic acid under conventional polyamide polymerization conditions using caprolactam.

25 Example 1 - Semifinished slab produced from polyamide according to the invention and reinforcing yarns

A series of trials was performed using a polyamide Al multifilament yarn described above, having a strand linear density of between 21 and 22 dtex and a tenacity of about 23 cN/tex. Such a multifilament was assembled, in a multiaxial weaving operation, with a continuous glass reinforcing yarn having a linear density of 600 tex. To validate the high melt flow of the matrix, multiaxial woven fabrics were produced from elementary layers, each being defined as below:

Elementary layer

ply 1: reinforcing yarn, -45° orientation

ply 2: reinforcing yarn, +45° orientation

5 ply 3: polyamide A1 (matrix) yarn, 90° orientation.

A laminated composite was then produced by placing several (between 2 and 10) elementary layers of the fabric obtained in a slab-shaped mold between the heated plates of a press, for a time of 1 to 3 minutes, under a pressure of between 1 and 20 bar and a temperature between 250 and 260°C (above the melting point of the polyamide A1). After cooling down to a temperature of 50-60°C, the composite was demolded. The reinforcement content was then between 60 and 70% by weight.

The high melt flow of the polyamide Al made it possible to achieve good impregnation of the reinforcement with the matrix without causing the reductions in mechanical properties or the fatigue strength problems observed with low-molecular-weight polymers. The mechanical properties in bending are compared with those of a thermoset-based composite obtained from the same reinforcing material and an epoxy resin in Table 1.

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Table 1: Polyamide/glass fiber composite slab

Table 1: Tolyamiac, grass 115				
	Tensile	Flexural	Elongation	
	strength	modulus	at break	
	(MPa)	(MPa)	(용)	
Epoxy matrix/glass	630.0	21 000	3.53	
fibers				
PA1 matrix/glass	517	21 000	3.26	
fibers				

The use of a reinforcement in continuous yarn form makes it possible to maintain high mechanical properties in

preferred directions. The fact of using the matrix in the form of a yarn provides, in addition to an economic advantage compared with the conventional solutions of powder coating or preimpregnation, easy handling and good control of the reinforcement content of the final composite.

Table 2 summarizes the mechanical properties obtained.

Table 2: Summary of the mechanical properties obtained

Table 2: Summary of the mechanical properties obtained				
	Units	Standard	PA A1/glass multiaxial fabric	
Degree of	o _o		65	
impregnation (p/p)				
Density			1.8	
Simple tension				
Stress at break	MPa	ISO 527	545	
Young's modulus	GPa	ISO 527	21.3	
Elongation	olo	ISO 527	2.76	
3-point bending				
Stress at break	MPa	ISO 14125	517	
Flexural modulus	GPa	ISO 14125	21	
Multiaxial shock				
Maximum force	DaN	ISO 6603-2	650	